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B5N

(54) **Production of a laminate**

(57) A method of producing a laminate comprises arranging a pair of sheets (1, 2), e.g. of glass, face-to-face and at an incline to the horizontal with a lower portion of the periphery of the sheets sealed in a substantially liquid tight manner by non-porous adhesive strip material (3-6) sandwiched between the sheets. Settable liquid resin material is poured between the sheets through at least one filling opening along an upper portion of the periphery of the sheets. When all the resin material has been introduced, the upper portion of the sheet periphery is sealed with the exception of air gaps (11, 12), the sheets (1, 2) are lowered to a horizontal position to enable air to be expelled through the air gaps (11, 12) and the liquid resin material is allowed to set.

FIG. 2

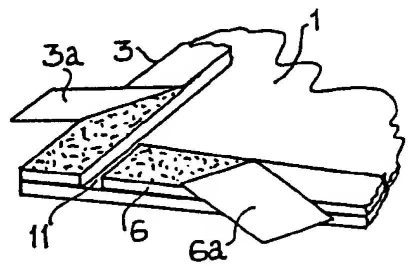

FIG. 4

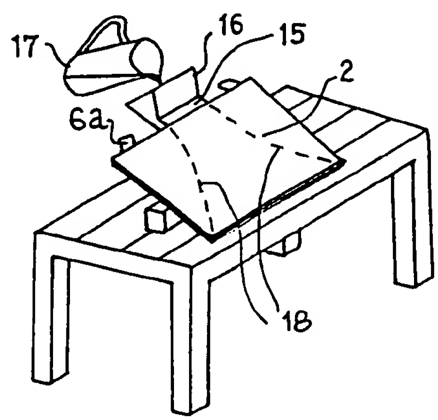

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FIG. 1

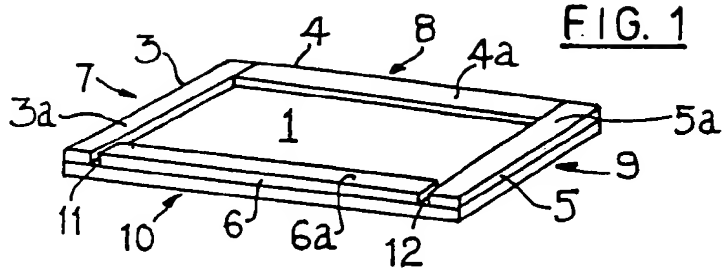


FIG. 2

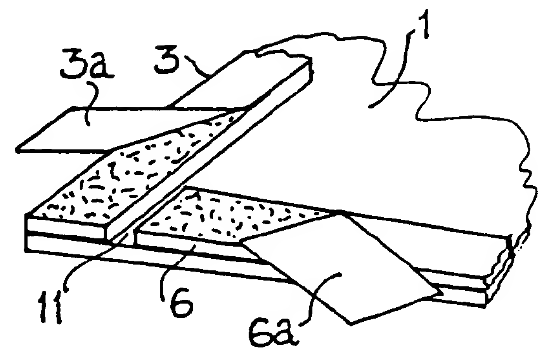


FIG. 3

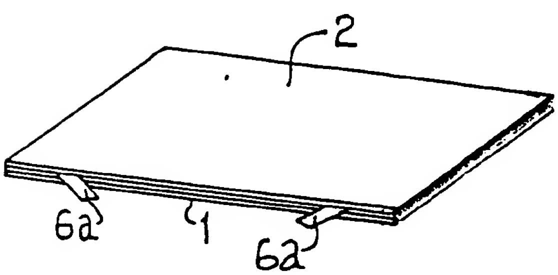
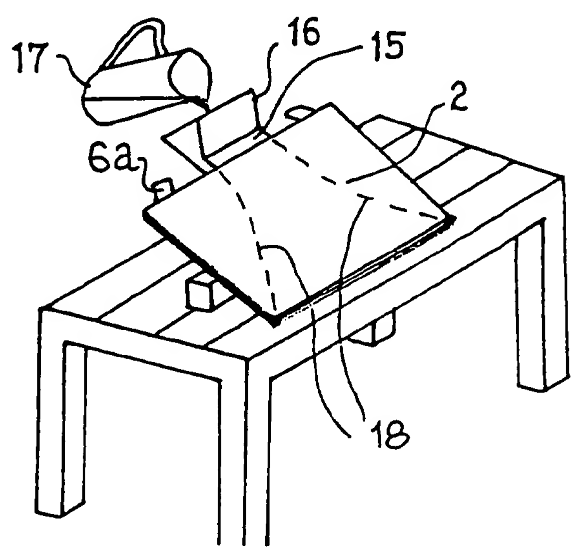


FIG. 4



SPECIFICATION

Production of a laminate

This invention r lates to a meth d of producing a laminate, e.g. a laminated glass structure comprising two or more sheets of frangible material, e.g. glass, b nded tog th r and t a laminate made by such a method. In particular, but not exclusively, the invention relates to the manufacture of safety glass.

According to the invention a method of producing a laminate comprises arranging a pair of sheets of frangible material, e.g. glass, face-to-face at an incline to the horizontal, the said sheets being sealed together in a substantially liquid tight manner around a lower portion of the periphery of the sheets using non-gas-permeable adhesive strip material sandwiched between the sheets, introducing a predetermined quantity of settable liquid resin material between the sheets at at least one filling region along an upper portion of the periphery of the sheets, sealing the sheets together in a substantially liquid tight manner around the said upper portion of the periphery of the sheets after the introduction of said predetermined quantity of liquid resin material so that the sheets are sealed around their entire periphery with the exception of air gaps formed therein at spaced locations on either side of the or each filling region, moving the sheets into a substantially horizontal position to enable the liquid resin material to spread evenly between the sheets and expel air from between the sheets through said air gaps, and allowing the liquid resin material to set to form a bonded laminate.

Preferably the said upper portion of the periphery of the sheets is sealed in said substantially liquid tight manner by adhesive strip material impervious to gas sandwiched between the said sheets.

The invention makes use of the phenomenon that the liquid resin material, introduced into the interspace between the sheets of frangible material, will move across the bottom sheet along a path of least resistance when the sheets are moved into their horizontal position. This path of least resistance is provided by the part of the bottom sheet which has already beenwetted or lubricated by the downward flow of liquid resin material as it is introduced between the previously inclined sheets. After flowing back along this path of least resistance, the liquid resin material gradually spreads outwards to provide an even thickness interlayer between the sheets. Air trapped between the sheets cannot pass through the adhesive strip material which acts as an air barrier and is therefore expelled, as the liquid resin material spreads towards the air gaps, through the air gaps. It will of course be appreciated that there may also be some minor outward spread of the liquid resin material during movement of the resin material back along said path of least resistance.

Suitably the sheets of frangible material are inclined to the horizonatal at an angle of from 15° to 65°, preferably from 20° to 40°, e.g. 30°, during introduction of the liquid resin material between the sheets.

Preferably the settable liquid resin material is prepared by mixing, e.g. hand mixing (as opposed to machine mixing), a liquid resin with one or more catalysts.

Conveniently the said gas-impervious adhesive strip material is flexible. Preferably the adhesive strip material is double-sided adhesive tape and is sandwiched between the sheets adjacent the periphery thereof. Conveniently the sheets are rectangular, both having substantially the same lengths and substantially the same widths. Preferably the sheets are sealed adjacent first, second and third sides of the sheets prior to the introduction of the settable liquid resin material between fourth sides of the sheets.

The sheets may be sealed by applying the adhesive strip to a face of one of the sheets along the first, second and third sides thereof, positioning the other sheet in face-to-face relationship with the said one sheet and removing a non-adhesive backing layer from the applied adhesive strip material so that the sheets are sealed together by the adhesive strip material. Adhesive strip material may also be applied along the fourth side of the said one sheet, the backing layer being removed therefrom after the introduction of the liquid resin material to enable sheets to be sealed along the fourth sides thereof prior to moving the sheets into their horizontal position.

Conveniently the liquid resin material is introduced between the sheets via at least one funnel device inserted between the sheets along said upper portion of the periphery of the sheets. The liquid resin material is normally transparent but may also be coloured or tinted.

Typically the said air gaps are sealed after all the air has been evacuated, e.g. naturally, from between the said sheets.

A multi-laminate structure may be manufactured according to the invention comprising more than two thicknesses of sheets of frangible material, e.g. glass, adhered together.

Embodiments of the invention will now be described, by way of example, with reference to the accompanying drawing, in which:-

Figures 1-4 are schematic representations showing various stages in the production of a laminate, e.g. a laminated glass structure, according to the invention.

A laminated glass structure is made by firstly selecting two smooth faced, rectangular glass sheets or panels 1, 2 (see Figures 1-4) having approximately the same length and width and typically als the same thickness as each other. Typically each glass panel 1, 2 has a surface area of appr ximat ly 1 m². The glass panels ar then thoroughly cleaned, e.g. with glass cleaner or in a machin wash.

One f the clean glass panels 1 is then laid in a horizontal position and strips 3-6 (see Figure 1) of at least substantially gas- (.g. air-) impervious (e.g. no porous), d uble-sided adhesive strip material having n n-adhesive backing layers *3a-6a* appli d thereto are adher d to the sm th upper fac of th glass panel 1 along its f ur sides 7-10. Th adhesive strip material is suitably in the form f flexible strip material, e.g. tape, but c uld b in th form f a n n-flexibl or even rigid strip. The adhesiv layers of the strip material preferably comprise pressure-sensitive adhesive coatings.

The strips 3-6 are butt jointed at the corners between the sides 7 and 8 and 8 and 9. However, the strip 6 is arranged so that small gaps 11 and 12, e.g. of approximately 2 mm width, are provided at its opposite ends adjacent the strips 3 and 5. The strip 6 may be pre-cut to a length slightly shorter than the distance between the strips 3 and 5 so that these gaps 11 and 12 are automatically formed when the strip 6 is laid in position. Alternatively the ends of the strip 6 may be cut, e.g. by a razor blade, after the strip has been adhered to the glass panel 1, to form the gaps 11 and 12. The opposite end portions of each of the non-adhesive backing layers *3a-6a* are then peeled back a short distance and folded over and outwardly, in the manner shown in Figure 2, to expose adhesive surfaces of the strips 3-6 at opposite ends thereof.

The other clean glass panel 2 is then positioned above the horizontal glass panel 1, in face-to-face relationship therewith, and is carefully lowered onto the strips 3-6 of adhesive strip material, taking care to align the sides of the two glass panels 1, 2, so that the strips 3-6 are sandwiched between the panels 1 and 2. The previously peeled back and folded over end portions of the backing layers *3a-6a* project outwardly from between the confronting panels 1 and 2. By gripping the folded over end portions of the backing layers *3a-5a*, the backing layers *3a-5a*, but not the backing layer *6a*, are then manually pulled away from the strips 3-5 and the glass panel 2 is firmly pressed against the revealed adhesive surfaces of the strips 3-5 (see Figure 3) to ensure good adhesion thereto. If there are any gaps present between the butt jointed strips 3, 4 and 4, 5 these gaps are sealed by means, for example, of mastic so that a liquid tight seal is formed between the glass panels 1 and 2 along the sides 7, 8 and 9.

The assembly of sealed glass panels 1 and 2 is then raised into an inclined position with its unsealed edge uppermost. Typically the panels are inclined at an angle of from 15° to 65°, preferably from 20° to 40°, e.g. 30°, to the horizontal. The unsealed edge of the glass panel 2 is carefully lifted from the underlying backing layer *6a* of the adhesive strip 6 and a broad but thin spout 15 of a funnel 16 is inserted between the now slightly spaced apart glass panel 2 and backing layer *6a* (see Figure 4) to permit previously prepared settable liquid resin material to be poured through the funnel downwardly between the glass panels 1 and 2.

The settable liquid resin material is preferably prepared by a so-called hand mixing process involving the hand mixing or stirring of a low viscosity liquid resin with at least one catalyst. A particularly preferred resin material is obtained by mixing, e.g. hand mixing, "Naftolan Resin S696" or "Naftolan Resin S700" with catalysts designated "K66", "K206" and "K73" (the designated resins and catalysts are manufactured by Deyussa AG, Geschaftsbereich Chemie, Frankfurt, West Germany, and distributed by Chemetall, Gesellschaft Technische Verfahren mbH Sparte Glas Reutervey 14, Frankfurt, West Germany. These particular resins are methacrylate resins and incorporate an ultra violet stabiliser. They are characterised by being of extremely low viscosity for such resin materials and are extremely clear (typically having a light transmittance of 92% compared with ordinary glass which has a light transmittance of from 80% - 90%.

The volume of resin material required to bond the glass panels 1 and 2 together is calculated by multiplying the surface area of one side of one of the glass panels 1, 2 by the desired interlayer thickness, normally 0.8 mm to 1.5 mm, required between the panels 1, 2. The calculated volume may be adjusted slightly to allow for shrinkage during setting of the resin material.

The predetermined quantity of selected resin "S696" or "S700" is then measured out by being slowly poured into a measuring beaker 17 and is allowed to degas sufficiently to ensure no micro bubbles are held in the basic resin. The actual rate of degassing is dependent on the temperature of the operating environment.

When the selected resin is absolutely clear, and has no trapped air therein, the catalysts are slowly poured into the resin and gently, but thoroughly, manually stirred, i.e. with the aid of a hand held stirrer. Care must be taken to ensure adequate mixing without generating air micro bubbles. If bubbles are generated during mixing, sufficient time must be allowed to elapse for them to dispense.

Catalysts "K206" and "K66" are added to the selected resin, suitably at a ratio of 100:1 by volume, and catalyst "K73" is added to the resin suitable at a ratio of 100:0.5 by volume. However, these ratios are only an approximate guide and can be substantially altered to affect clarity, strength and curing rate of the resin material.

The curing rate of th liquid resin material with hand mixing is influenced by the operating temperature, which should ideally be between 15°C and 25°C. At temperatures under 15°C th rati of resin and catalyst must b altered to effect proper curing. At tempertatures below 1°C the hand mix ratio of resin and catalysts must be altered and further curing can only be effected by applying heat to the filled laminated glass for a minimum of 120 minutes. Thicker resin layers exceeding 1.5 mm, such as required in heavily patterned glass and wh re operating temperatures are high should be cooled by air to prevent fracture of the resin or glass during curing.

By using liquid resin having a relatively low viscosity, a homogenous mixture of liquid resin and catalyst(s) can be btained by a hand mixing process - i.e. by the use of a hand held stirr r - and does not involve the use of expensive mixing machinery. This means that an acceptable standard of laminate can be produced with minimal capital expenditure for machinery. Furtherm re the relatively low viscosity of the resin/catalyst mixture enables any bubbles (e.g. microbubbles) cr ated by the hand mixing to dissipate freely without the use of additi nal degassing equipment (e.g. h ating equipment or a vacuum pump).

The measured quantity of degassed resin and catalysts is then poured through the funnel 16 between the inclined glass panels 1, 2, great care being taken to ensure a slow even pour into the centre of the interspace between the panels. If pouring occurs at too fast a rate curing may be affected and may result in visual shrinkage faults.

In Figure 4 the manner in which the resin material fills up the panel interspace by gravity flow is shown schematically by dashed lines 18. In particular it will be noticed that the liquid resin material flows downwardly and outwardly, with the upper corners of the interspace, in the region of the air gaps 11 and 12, left substantially unfilled.

After the predetermined, measured amount of liquid resin material has been introduced into the interspace between the glass panels 1 and 2, the funnel 16 is withdrawn ensuring that little or no resin material drips over the backing layer *6a* of the adhesive strip 6. Then the folded over end portions of the backing layer *6a* are gripped, the backing layer *6a* is carefully, manually, pulled away from the strip 6 and the glass panel 2 is pressed firmly against the revealed adhesive surface of the adhesive strip 6 to ensure good adhesion thereto. At this stage the glass panels will be slightly bulging apart, and the liquid resin material will only partly fill the interspace between the panels 1 and 2.

The assembly of glass panels 1 and 2 is then lowered to a horizontal position and air is allowed to evacuate naturally through the gaps 11 and 12. It is important to ensure that the glass panels are positioned horizontally as soon as possible after the liquid resin material has been poured into the interspace between the panels 1 and 2 so that the resin material is still in a flowable state.

When the panels 1 and 2 are in the horizontal position, the weight of the upper glass panel 2 causes the liquid resin material to spread out to provide an even thickness interlayer between the glass panels. Initially the liquid resin material flows back over the lower glass panel 1 along a path of least liquid flow resistance, i.e. over the part of the panel 1 previously "wetted" or "lubricated" by the resin material as it was poured into the panel interspace. After a short time the liquid resin material meets the strip 6 and then gradually spreads outwardly towards the unfilled corners of the panel interspace thereby expelling air from the interspace through the air gaps 11 and 12. The strips are non-porous and prevent the air from passing therethrough. When the panel interspace is completely filled with an even thickness of resin material and the air is fully evacuated from between the glass panels 1 and 2, typically after approximately 2 minutes, the gaps 11 and 12 are sealed, e.g. with mastic, and the resin material is allowed to cure (with the assembly still in a horizontal position) for approximately 3 to 6 hours at normal room temperature. If some air bubbles become trapped between the glass panels 1 and 2 and will not evacuate naturally, they may be extracted by known means, e.g. with the use of a syringe (not shown) or vacuum pump (not shown), prior to sealing of the gaps 11 and 12.

It will be appreciated that, especially in the case of extra long glass sheets (e.g. having long sides 8 and 10), it may be necessary to have a number of spaced apart filling points along the upper edge of the periphery of the glass sheets. For example two or more funnels 16 could be employed for introducing liquid resin material into the interspace between the glass sheets. In this case, however, it is necessary to provide at least one air gap in the peripheral seal between each pair of adjacent resin filling points.

In this specification the purpose of the adhesive strip material sandwiched between the glass sheets around the periphery thereof is to block the passage of the liquid resin material therethrough when the latter is poured between the sheets and during the setting of the resin material. In addition, the strip material is impervious to gas at least to the extent that air expelled from between the glass sheets as the liquid resin material spreads between the sheets will not pass through the strip material but will be forced to pass out through the air gaps 11 and 12. A particularly suitable adhesive strip material is a polyurethane tape known as "Normount V2500" (a trade name of the U.S. company Norton Performance Plastics). This particular tape has the following physical characteristics:

Colour : Black
Hardness (Shore A) : 45+/-15
Force to compress (kg/cm) : max 2.5, min 0.8
Compression deflection (min % loss from original height) : 0.5
Water absorption (% by volume) : 2 max
P el adhesion (kg/cm) : 1.0
Elongation (% min) : 250
Service Temperature (°C) : −35 to +95
Paint staining : 0

Such tap possesses th required qualities for acting as a barrier to the liquid resin material and air during the time that air is being expelled from the air gaps.

CLAIMS

1. A method of producing a laminate comprises arranging a pair of sheets of frangible material, face-to-face at an inclin to the horizontal, the said she ts being sealed together in a substantially liquid tight manner around a l wer porti n f the periphery of the sh ets using adhesive strip material, which is at least substantially impervious to gas passing through, sandwich d between the sheets, intr ducing a predetermined quantity of settable liquid resin material between the sheets at at least one filling region along an upper portion of the periphery of the sheets, sealing the said sheets together in a substantially liquid tight manner around the said upper portion of the periphery of the sheets after the introduction of said predetermined quantity of liquid resin material so that the sheets are sealed around their entire periphery with the exception of air gaps formed therein at spaced locations on either side of the or each filling region, moving the sheets into a substantially horizontal position to enable the liquid resin material to spread evenly between the sheets and expel air from between the sheets through said air gaps, and allowing the liquid resin material to set to form a bonded laminate.

2. A method according to claim 1, in which the said upper portion of the periphery of the sheets is sealed in said substantially liquid tight manner by non-gas-permeable adhesive strip material sandwiched between the sheets.

3. A method according to claim 1 or 2, in which the sheets of frangible material are inclined to the horizontal at an angle of from 15° to 65° during introduction of the liquid resin material between the sheets.

4. A method according to claim 3, in which the said angle is from 20° to 40°.

5. A method according to any of the preceding claims, in which the said adhesive strip material is flexible.

6. A method according to any of the preceding claims, in which the adhesive strip material is double-sided adhesive tape.

7. A method according to any of the preceding claims, in which the sheets are rectangular, both having substantially the same lengths and substantially the same widths.

8. A method according to claim 7, in which the sheets are sealed adjacent first, second and third sides of the sheets prior to the introduction of the settable liquid resin material between fourth sides of the sheets.

9. A method according to claim 7 or 8 when dependent upon claim 6, comprising sealing the said lower portion of the periphery of the sheets by applying the adhesive strip to a face of one of the sheets along the first, second and third sides thereof, positioning the other sheet in face-to-face relationship with the said one sheet and removing a non-adhesive backing layer from the applied adhesive strip material so that the sheets are sealed together by the adhesive strip material.

10. A method according to claim 9, in which double-sided, gas-impervious adhesive strip material is also applied along the fourth side of the said one sheet, the backing layer being removed therefrom after the introduction of the liquid resin material to enable sheets to be sealed along the fourth sides thereof prior to moving the sheets into their horizontal position.

11. A method according to any of the preceding claims, in which the liquid resin material is introduced between the sheets via at least one funnel device inserted between the sheets along said upper portion of the periphery of the sheets.

12. A method according to any of the preceding claims, in which the liquid resin material is transparent.

13. A method according to any of claims 1 to 12, in which the liquid resin material is coloured or tinted.

14. A method according to any of the preceding claims, in which the said air gaps are sealed after all the air has been evacuated, e.g. naturally, from between the said sheets.

15. A method according to any of the preceding claims, in which the said settable liquid resin material is prepared by hand mixing a relatively low viscosity liquid resin with at least one catalyst.

16. A method according to claim 15, in which the said liquid resin has a light transmittance of approximately 92%.

17. A method according to claim 15 or 16, in which the said liquid resin comprises a methacrylate resin.

18. A method according to any of the preceding claims, in which the said settable liquid resin material includes an ultra violet stabiliser.

19. A method of producing a laminate substantially as herein described with reference to, and as illustrated in, Figures 1 to 4 of the accompanying drawing.

20. A laminate produced by a method as claimed in any of claims 1 to 19.

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